

# PATENT SPECIFICATION

DRAWINGS ATTACHED

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## COMPLETE SPECIFICATION

### Improvement in or relating to Drying Polyamide Granulate

We, INVENTA, A.G. FÜR FORSCHUNG UND PATENTVERWERTUNG, Haldenstrasse 23, Lucerne, Switzerland, a body corporate organised under the laws of Switzerland, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

As a result of the development of continuous production techniques for the manufacture of a polyamide granulate it has been necessary to devise new processes of drying the granulate. Vacuum driers have been proposed but the presence of even a very small quantity of air in such a drying process has, under certain conditions, a harmful effect and this therefore makes an absolute vacuum essential.

According to the present invention there is provided an economically advantageous process for the continuous drying of polyamide granulate at temperatures below the melting or softening points of the polyamide. In this process the wet granulate slides downwards through a drying tower which comprises at least two sections, each section having greater diameter and less height than the section immediately below it, while a gas inert to the polyamide, and having a temperature sufficient to remove the water, is introduced into the bottom of each section and flows upwards.

The invention also provides an apparatus for drying polyamide granulate which comprises a drying tower having an inlet for the polyamide at the top and an outlet at the bottom, the tower having at least two sections each section having greater diameter and less height than the section immediately below it. There is provided means for introducing the drying gas which is inert to the polyamide at the bottom of each section and for withdrawing the gas from the top of the tower,

and there is also provided means for heating the drying gas before it is introduced into the tower.

The preferred inert drying gas is pure nitrogen, which acts simultaneously as a protective gas, as a conveying or entraining medium for the water to be removed, and as a heat-transmitting medium. The transmission of heat to the granulate, which may be in the form of chips is considerably better, and thus much shorter drying times are possible, than in the case of drying in vacuo. In principle, the process is a continuous counter-current drying process, the chips being introduced at the top of the drying tower and leaving it again at the bottom, while dry nitrogen heated to the drying temperature, which must be below the melting or softening point of the polyamide, is blown in at the bottom and emerges at the top charged with moisture. The wet chips are dried in at least two sections. The main quantity of water is removed in the preliminary drying stage, which takes place in the top section of the tower, and, consequently, the main proportion of the quantity of heat required must also be introduced here, i.e. a large quantity of nitrogen is necessary in this stage. So that the gas velocity and the drop in the pressure of the nitrogen should not become too great, this section of the drying tower has a small height with a relatively large diameter. As a uniform flow of the chips does not matter in this section the adhering water and the majority of the water contained in the chips can be removed relatively easily, any irregularities being adjusted in the last stage of the drying process, which takes place in the lowest section of the tower.

In principle, the drying can be carried out at any temperature below the melting or softening point, but the temperature must not be so high that moisture condenses onto the chips when they are withdrawn from the

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bottom of the tower. The temperature must also be high enough to promote drying. The preferred temperature range is therefore between 50° C. and 200° C.

5 Generally, it is sufficient if drying is carried out in two stages, i.e. if the first stage is followed immediately by the final stage, but, in given circumstances, it may be advantageous to interpose one or more intermediate stages  
10 between the first and last stage, in such manner that the height or depth of the layer of polyamide chips increases from stage to stage and the diameter thereof decreases. In general, nitrogen is used as drying gas, but  
15 in principle any gas which is inert towards the polyamide under the conditions of the process may be employed.

The drying gas may, of course, be circulated. So that too much steam does not accumulate in the gas, part of the stream is separated directly after leaving the top of the tower, so that the majority of the water can be eliminated and separated. The gas is there-upon re-heated to drying temperature and introduced into the bottom of the lowest section. In this section, the final drying is effected by the gas freed from water. The gas then flows up through the column and enters the higher section, which, in the case  
20 of a tower comprising only two sections, will be the section where the preliminary drying occurs.

The entire drying plant is advantageously operated at a small excess pressure, so that  
25 no atmospheric air can penetrate. A small quantity of fresh gas is blown through the plant for this purpose. A corresponding quantity of gas issues through the chip-charging tube and also prevents the penetration and entrainment of atmospheric air at this point. The chips do not reach the temperature  
30 required for driving out the last traces of water until the end of the drying process and are at this temperature for only a short time, whereby very careful drying is made possible.

Contrary to the views heretofore held, it has been found that according to the process of this invention, polyamides can also be dried with air if the drying is carried out at temperatures at which yellowing does not yet take place, generally below 100° C. Unless very extreme drying (a final water content of 0.1%) is to be carried out, troublesome yellowing of the chips does not occur.

55 The invention will be described in detail with reference to the accompanying drawing.

The chips pass through a tube 1 into a drying plant, travel through section I and immediately after through section II and are discharged at 2. The drying gas is circulated by means of a fan 3. The main quantity flows through pipes 4 and 5 to gas heater 6 and is fed from here through a pipe 7 and an annular passage 8 to the bottom of the first section of the drying tower. After flowing  
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through this section, the gas arrives back at the fan again through a pipe 9. A partial flow passes through the pipes 4 and 10 into a heat exchanger 11 and thereupon into a cooler 12. Here, the majority of the water is separated out by condensation and flows, together with the gas, through a pipe 13 into a separator 14, is separated from the gas and drawn off through a pipe 15. The gas flows through a pipe 16 to the other end of the heat-exchange surface of the exchanger 11 and thereupon through a pipe 17, a heater 18, a pipe 19 and an annular passage 20 into the bottom of the second section of the drying tower, flows through this section and then through the first section and arrives back at fan 3 through the pipe 9. A small stream of fresh gas is fed into the drying plant through a pipe 21 and a corresponding quantity of circulating gas leaves the plant through the pipe 1.

#### EXAMPLE 1

Poly-ε-caprolactam chips having a grain size of 2.5 mm are dried in a plant such as that described above. The first section has a layer depth of 600 mm with a diameter of 200 mm and the second section has a diameter of 150 mm and a length of 1800 mm. 2 kg/h of dry chips are discharged at the bottom through the discharge means, which corresponds to a time of residence of about 16 hours. The quantity of nitrogen supplied to the first section is 40 cu.m./h at 125° C. and that supplied to the second section is 8 cu.m./h at 110° C. The gas supplied to the second stage heater is previously cooled to 20° C. in the cooler and the extracted water is separated. The quantity of fresh nitrogen is 0.7 cu.m./h. The chips dried in this way have a water content of 0.11%.

#### EXAMPLE 2

Poly-ε-caprolactam chips are dried in the plant as in Example 1. The quantity of chips discharged is 1.4 kg/h. The quantity of gas supplied to the first section is 30 cu.m./h of nitrogen at 125° C. and that supplied to the second section 10 cu.m./h at 130° C. This latter quantity of gas is previously cooled to 5° C. and the extracted water is separated. The quantity of fresh nitrogen is 0.7 cu.m./h. The chips have a final water content of 0.04%.

#### EXAMPLE 3

Poly-ε-caprolactam chips are dried in a plant such as that used in Example 1. The quantity of chips discharged is 1 kg/h. The quantity of air supplied to the first section is 25 cu.m./h at 70° C. and the quantity of air supplied to the second section is 10 cu.m./h at 82° C. The air is sucked in from the atmosphere through a filter and is recycled as described above, and a small quantity of the air leaving the first section is blown off again into the atmosphere at the top of the drying  
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- plant. The air recycled from the first section which is intended for the second section is compressed to 10 atmospheres excess pressure and cooled to 20° C., the water is separated and the air is again expanded. The final water content of the chips is 0.12%. Injection-moulded parts made therefrom do not exhibit any impairment of the properties of the chips whatsoever.
- 10 WHAT WE CLAIM IS:—
1. A process for the continuous drying of polyamide granulate at temperatures below the melting or softening point of the polyamide in which the wet granulate slides downwards through a drying tower comprising at least two sections, each section having a greater diameter and less height than the section immediately below it, while a gas inert to the polyamide and having a temperature sufficient to remove the water is introduced into the bottom of each section and flows upwards.
  2. A process as claimed in Claim 1, in which nitrogen is used as the drying gas.
  - 25 3. A process as claimed in Claim 1, in which air is used as the drying gas.
  4. A process as claimed in any preceding claim in which the drying is carried out at temperatures from 50° to 200° C.
  - 30 5. A process as claimed in any preceding claim carried out in a drying plant in which the drying gas is circulated, the water extracted from the granulate being separated by cooling from part of the stream of drying gas emerging from the top of the drying tower and, after reheating, the water-free drying gas is introduced to the tower at the bottom of the lowest section.
  - 35 6. A process as claimed in any preceding claim in which the drying tower comprises two sections.
  7. An apparatus for drying polyamide granulate comprising a drying tower having an inlet for the polyamide at the top and an outlet at the bottom, the tower having at least two sections, each section having greater diameter and less height than the section immediately below it, there being means for introducing a drying gas inert to the polyamide at the bottom of each section and means for withdrawing the gas from the top of the tower, there also being means for heating the drying gas before being introduced into the tower.
  8. An apparatus as claimed in Claim 7, in which there are means for circulating the drying gas and also means for drying at least part of the drying gas emerging from the top of the tower, said drying means comprising a cooler and separator.
  9. An apparatus as claimed in Claim 7 or 8 in which the drying tower comprises two sections.
  10. A process for the continuous drying of polyamide granulate substantially as herein described with reference to any one of the examples.
  11. An apparatus for drying polyamide granulate substantially as herein described with reference to the accompanying drawing.
  12. Polyamide granulate whenever dried by a process as claimed in any one of Claims 1 to 6 or 10.

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